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Measurement of some nitride layers by nuclear reaction analysis technique

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Abstract : Nuclear reaction analysis (NRA) technique in measuring the thickness of nitride layer up to 9030 Å has been investigated using 2 MeV deuterons. A calibration curve for future measurements of thickness of thin films of silicon nitride is provided. The technique is compared with Rutherford backscattering spectrometry (RBS) technique and is found to be excellent for relative measurements of layer thicknesses.

Keywords : Nitride layer thickness, nuclear reaction analysis, (d,p) reaction

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1. Introduction

Nuclear reaction analysis method has proved to be a useful one in depth profiling of different nuclides in various materials allowing evaluation of the composition and thickness of nitride and oxide films on bulk elemental surfaces [1–4]. During the past few decades, the technique has been developed for near surface analysis [5–10]. Side by side progress of RBS technique for the same purpose also continued. This can suitably be applied for semiconductor fields specially for silicon nitrides. Amongst other modern surface analytical techniques such as secondary ion mass spectrometry, auger electron spectrometry and X-ray fluorescence, these two techniques are also of considerable interest.

These two methods have been applied for oxide layer measurements in our previous paper [11]. As techniques for depth profiling, the role of these methods has been adjudged in that work. Whether a similar role is maintained for nitride layers also, is a subject of interest. The present work has been undertaken to investigate that.

All the samples used in this work, are silicon nitrides. Silicon nitride is a good insulator possessing a wide scale application in semiconductor devices, specially in the electronic components. The nitride layers of the present investigation are made on bulk silicon substrates. Accurate measurement of the thickness of the layer is necessary for its various application.

On these samples the RBS technique of depth profiling has already been applied by us [12]. The present work reports the results of thickness measurements by adopting NRA technique. A comparative study of the two methods has also been made in this work as it was felt that this sort of study would give us more insight about the applicability of the methods for the purpose of depth profiling.

2. Choice of the reaction

In the present work, the (d,p) reaction has been chosen because the reaction has its general applicability and its routine nature of analysis. It provides an extremely useful method of analysis of the elements having $z = 4$ to 17 for which case the Q-values are positive. (The Q-value for the reaction $^{14}N(d,p_o)^{15}N$ as measured by Piskor and Schaferlingova [13,14] is 8608 ± 0.061 keV). Therefore using the method, multi-elemental analysis is possible; of course, the reaction cannot be used for elements having $z > 17$ because Coulomb barrier effect reduces the sensitivity of the technique beyond the acceptable limits [15].

The cross sections for the different reactions induced by deuterons on ^{14}N, are reported in the literature [16]. It is known [1] that plateaus in the cross sections occur for $^{14}N(d,p_1 + p_2)$, $^{14}N(d,p_3)$, $^{14}N(d,p_5)$ and $^{14}N(d,\alpha)$ reactions for deuteron energies between 0.95 to 1.3 MeV. In addition, many reactions on the silicon substrates also occur together with it. But experimentally determined spectrum guided us to select the ground state peak as the only suitable one for the analysis. Excepting this peak, the other peaks were contaminated by groups arising from silicon substrate and hence could not be considered for analysis in the work. E_d was chosen as 2 MeV because this gives reasonable sensitivity and is well suited for the analysis.

3. Principles

When a beam of deuteron ions having sufficient energy to overcome the Coulomb barrier strikes a thin and single element target (Figure 1a), then nuclear reactions may occur. A series of peaks may be observed in the yield/energy spectrum (Figure 1b) which depict different possible excited states of the daughter nuclei. The energy of the light particles can be calculated using standard kinematic consideration [17].

The unknown peaks may be identified using a calibration curve showing the emitted particle energy and peak channel number. The usual way to reveal the depth profiles from the

spectrum is to create a depth scale from stopping power tables or from the parametrized stopping powers including the effect of any detector foil.

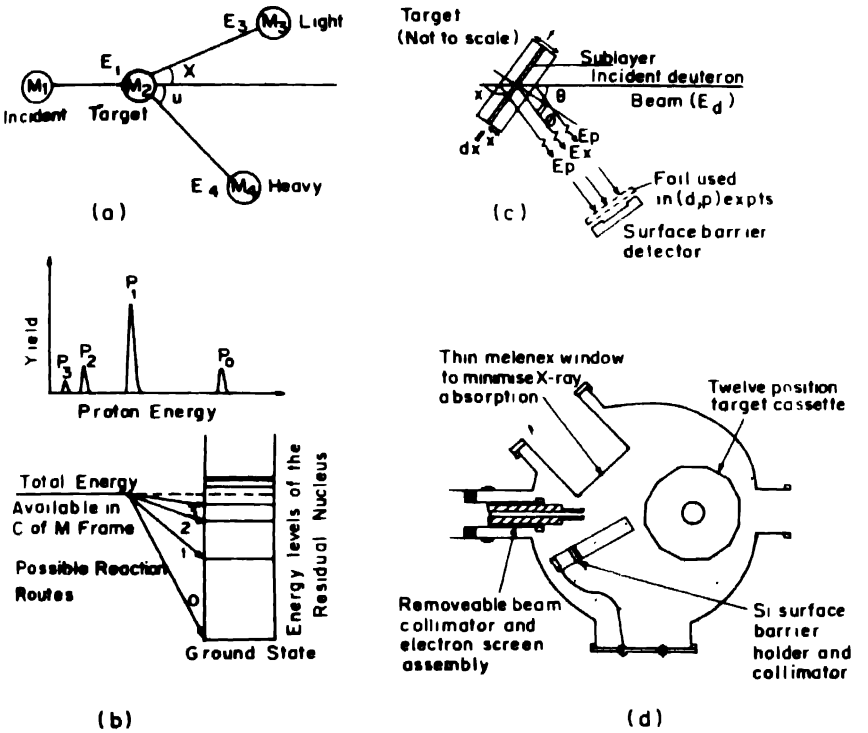

Figure 1. (a) Mechanism of a nuclear reaction. (b) A general yield/energy spectrum of emergent particles detected at the detector. (c) Position of the detector with respect to the incident deuteron beam (d) Schematic diagram of a general purpose nuclear analysis target chamber

4. Experimental

Nitride layer thicknesses of the samples cited in Table 1, are evaluated in the work using $^{14}N(d,p_0)^{15}N$ reaction. Deuterons produced from the 3 MV Dynamitron accelerator at Birmingham Radiation Center, University of Birmingham, were used as projectiles for the experiment. The geometries considered in the work are similar to that of our previous work [11] Following the previous work, only the angle 135° has been considered here (Figure 1c).

The experiment has been done on 10 different samples, all of which were Si_3N_4. These samples were made of nitride layers on bulk silicon substrates.Three samples were supplied by Plessey Ltd and the rest seven were provided by GEC Research Laboratories (Hirst Research Centre). The quoted nitride layer thicknesses of the samples are mentioned in the Column 4 of Table 1. The uncertainty of the sample thickness (not mentioned in the table) was around 8%.

As shown in Figure 1d, the deuteron beam was allowed to impinge on the samples mounted on the target holder (cassette) at 30° apart from each other carefully in such a way

that the incoming beam strikes these sides of the samples normally. The target chamber and the beam tube were evacuated by vacuum pumps to 10^{-6} Torr. The targets were striken by the incident beam of deuterons one after another normal to the plane of the target surface.

Table 1. Results of NRA and RBS measurements of layer thicknesses

Sl no	Name of the source	Composition	Quoted layer thickness (A)	Integrated area under $^{14}N(d,p_0)^{15}N$ peak	Calculated layer thickness from RBS (Å)
1	Plessey	Si_3N_4	991	779 ± 37	1000 ± 30
2	Plessey	Si_3N_4	1961	1541 ± 49	1990 ± 60
3	Plessey	Si_3N_4	9030	6550 ± 200	1000 ± 30
4	GEC	Si_3N_4	630	495 ± 27	660 ± 30
5	GEC	Si_3N_4	960	745 ± 35	970 ± 30
6	GEC	Si_3N_4	1200	943 ± 37	1210 ± 30
7	GEC	Si_3N_4	2180	1713 ± 52	2160 ± 60
8	GEC	Si_3N_4	2320	1824 ± 49	2330 ± 60
9	GEC	Si_3N_4	3130	2460 ± 59	3150 ± 90
10	GEC	Si_3N_4	4000	3144 ± 44	4030 ± 120

All the samples were made of nitride layers on bulk silicon substrates

A surface barrier detector with depletion layer 700 μm was placed at the said angle to the incident beam direction to detect the emitted particles. A 25 μm aluminum foil was placed just in front of the detector to stop the unwanted scattered deuterons. This thickness was chosen so as to be enough to stop the elastically scattered deuterons; besides it should be thin enough to allow the expected reaction products to pass through it. The beam current used in the experiment was 100 nA and integrated charge was 30 μC.

The process of obtaining the spectrum has been described elsewhere [11]. Signals from the detector were fed to an Ortec 125 pre-amplifier before it was fed to an Ortec 472A main amplifier by a long cable at counting station. The resulting pulses were passed in turn to a Hewlett Packard (HP) 5416B ADC and 2100 HP Computerized Multichannel Analyzer for the spectrum storage and display. The spectrum thus obtained was then stored on the disk and then copied to the magnetic tape for analysis.

The course gain and fine gain of the main amplifier were 200 and 0.9 respectively

5. Results and discussion

A representative spectra obtained in the present experiment is shown in Figure 2 where the group labelled as $^{14}N(d,p_0)^{15}N$ is the highest energy group and is the only interesting one Other groups labeled as (d,p_1) and (d,p_2) were contaminated by Si groups. They could not be separated and hence were avoided in the work.

The variation of the integrated areas with respect to the quoted nitride layer thicknesses of the samples has been shown in Figure 3a. Following previous work [11,18], here we have

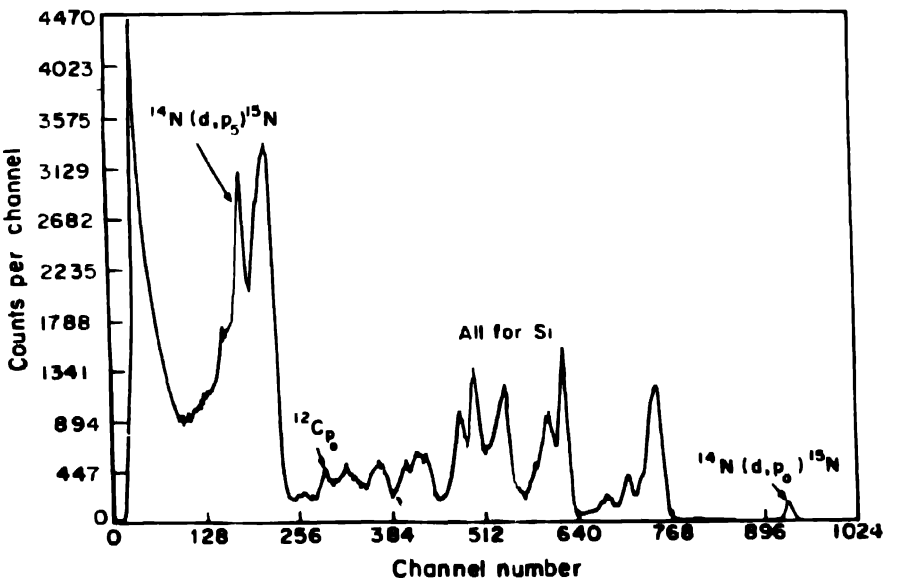

Figure 2. Energy spectrum of protons emitted during the deuteron bombardment of the Si_3N_4 sample having quoted layer thickness 2180 Å

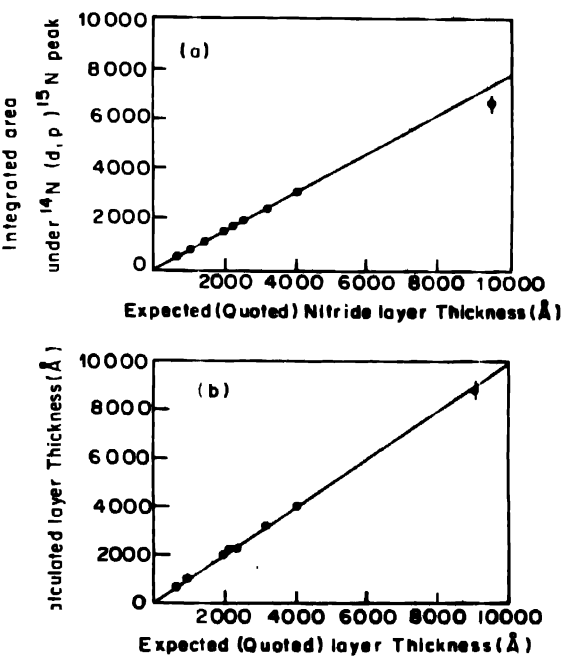

Figure 3. (a) Variation of expected layer thicknesses *versus* integrated area under $^{14}N(d,p_0)^{15}N$ peak initiated by the 2 MeV deuteron beam on the different Si_3N_4 samples having quoted layer thicknesses shown in Table 1. (b) Variation of calculated layer thicknesses for the different samples mentioned in Table 1 *versus* expected layer thickness from RBS analysis.

assumed that the cross section values do not vary appreciably over the range of energy loss of the incident particles in the layer being investigated. Under this condition, the total reaction yield may give a measure of the number of atoms present and the energy distribution may then provide a measure of the depth profile.

Among some other factors, the peak area also depends on the cross section which is not well known. Hence, we cannot get absolute thickness using this curve, rather the relative values of thickness may only be obtained. On the other hand, RBS measurements done previously by us on the same samples [12] prove its suitability (please see column 6 of Table 1 and Figure 3b) for determining the absolute thickness of the layer as the slope of the graph is 1 showing the variation of calculated layer thicknesses with respect to quoted layer thicknesses. However, for a much thicker layer, RBS technique would face difficulty in extraction of depth profiles, where (d,p) reaction analysis method may still be useful. It is worth mentioning here that NRA is a nondestructive technique and a quick process, the analysis time being only around 10 minutes per sample. The method is a better one than secondary ion mass spectrometry and AUGER at least in one aspect *e. g.*, unlike these methods the NRA method is free from artifacts inherent in methods involving sputtering.

6. Conclusions

In the present work, the $^{14}N(d,p)^{15}N$ reaction has been studied to investigate its suitability as a process of depth measurements. The technique is found to be excellent for relative measurements of layer thicknesses. A calibration curve for future measurements of thickness of thin films of silicon nitride is provided in the work. Although the NRA technique using $^{14}N(d,p)^{15}N$ reaction cannot give us the absolute measure of depth which RBS technique can provide, yet NRA technique can be employed for a thicker layer where RBS technique would fail.

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